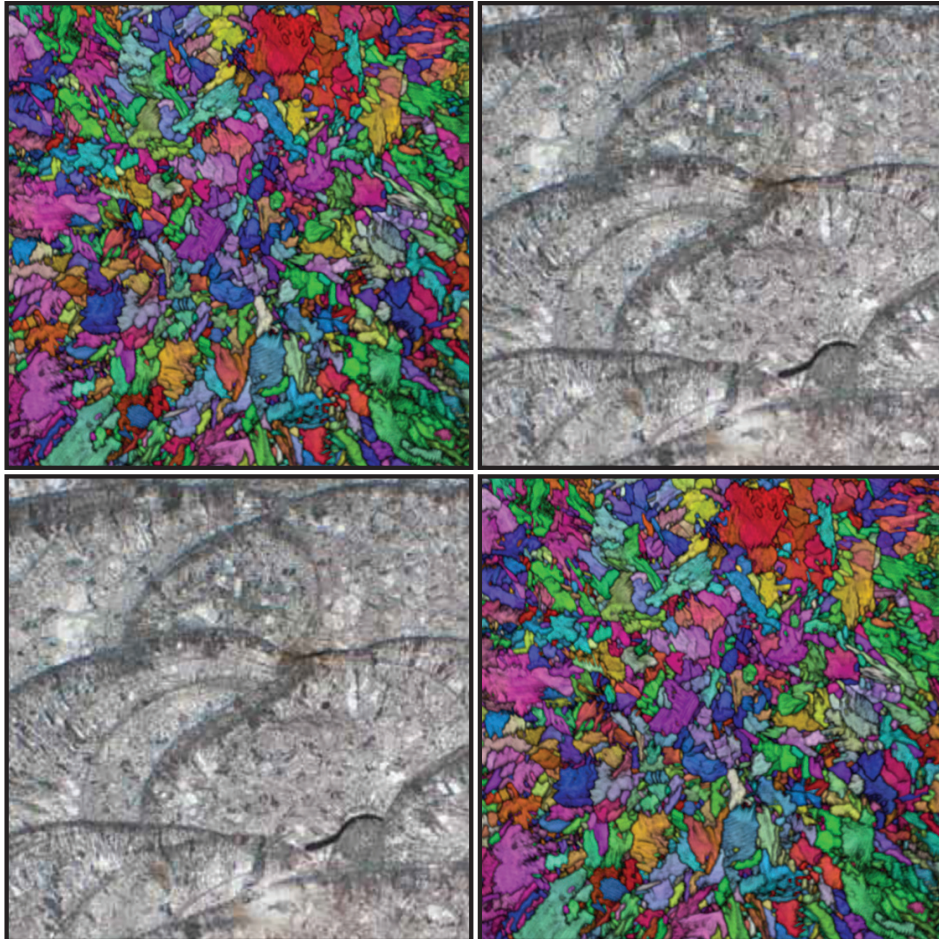# Advanced Qualification of Additive Manufacturing Materials

using in situ sensors, diagnostics and modeling

AGENDA

La Fonda on the Plaza – Santa Fe
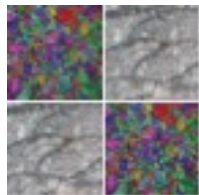# July 20 and 21, 2015

Welcome to our workshop on Advanced Qualification of Additive Manufacturing Materials using in situ sensors, diagnostics and modeling.

Workshop scope: Additive manufacturing (AM) offers distinct advantages over conventional manufacturing processes including the capability to both build and repair complex part shapes; to integrate and consolidate parts, and thus overcome joining concerns; and to locally tailor material compositions as well as properties. A variety of fields such as aerospace, military, automotive, and biomedical are employing this manufacturing technique as a way to decrease costs, increase manufacturing agility, and explore novel geometry/functionalities. To increase acceptance of AM as a viable processing method, pathways for qualifying both the material and the process need to be developed and, perhaps, standardized.

Our goal: To define opportunities and research gaps within AM and to engage the broader scientific/engineering community to discuss future research directions. In light of this, the organizing committee thanks you for attending this workshop in Santa Fe, NM - "Advanced Qualification of AM Materials – using in situ sensors, diagnostics, and modeling". Our invited speakers from universities and research centers, both US-based and Europe-based, will provide updates on their advanced qualification strategies or will present process-microstructure-property-performance results that motivate the need for an advanced qualification methodology. Talks over the course of the 1.5-day workshop will be mixtures of metals, non-metals, experimental, and modeling. Included in this Gordon style workshop are panel discussions with the invited speakers and the audience.

We hope that this workshop meets and surpasses your expectations, and that many projects stem from these one and a half days in July.

Advanced Qualification of Additive Manufacturing Materials *using in situ sensors, diagnostics and modeling*
July 20 & 21, 2015

Speakers

Suresh Babu - University of Tennessee

Mark Benedict - Air Force Research Laboratory

Kevin Chou - University of Alabama

Christian Leinenbach - EMPA Switzerland

Lyle Levine - NIST

Jyoti Mazumder - University of Michigan

Kalman Migler – NIST

Pan Michaleris - Penn State University

Todd Palmer - Penn State University

Natalie Rudolph - University of Wisconsin, Madison

Hans-Joachim Schmid - Universität Paderborn, DE

Dr. Chantal Sudbrack - NASA

Prof. Chris Tuck - University of Nottingham, GB

Organizing Committee

Alexander Balatsky

John Carpenter

Marianne Francois
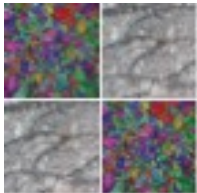
Rusty Gray

Terry Holesinger

Boris Maiorov

Nate Mara

Rick Martineau

Alex Mueller

Toni Taylor

David Teter

Dan Thoma

Advanced Qualification of Additive Manufacturing Materials *using in situ sensors, diagnostics and modeling*

July 20 & 21, 2015

Monday, July 20th 2015 – La Fonda on the Plaza – Santa Fe, New Mexico
7am – 7:30 pm

Venues:	Breakfast – Santa Fe Room
	Morning & afternoon sessions and Panel Discussion - New Mexico Meeting Room
	Lunch - La Terraza
	Poster Sessions - Santa Fe Room

|	
7:00 — 8:00	Registration in The Portal *and*
	Breakfast - provided by La Fonda on the Plaza
8:00 — 8:10	Introduction to Goals of Workshop –David Teter and Alexander Balatsky
8:10 — 8:50	Pan Michaleris
8:50 — 9:30	Hans-Joachim Schmid
9:30 — 10:10	Kevin Chou
10:10 — 10:30	*Break - with refreshments*
10:30 — 11:10	Mark Benedict
11:10 — 11:50	Natalie Rudolph
11:50 — 1:20	*Break for Lunch at La Terraza - provided by La Fonda on the Plaza*
1:20 — 2:00	Christopher Tuck
2:00 — 2:40	Chantal Sudbrack
2:40 — 3:20	Todd Palmer
3:20 — 4:00	Lyle Levine
4:00 — 4:15	*Break - with cocktails, chips and salsa*
4:15 — 5:45	Panel Discussion
5:45 — 7:30	Poster Session
	Light dinner hors-d'oeuvres and cocktails during Poster Sessions hors-d'oeuvres provided by La Fonda on the Plaza
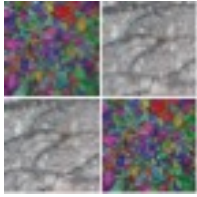
Tuesday, July 21st 2015 – La Fonda on the Plaza – Santa Fe, New Mexico
7:15 am — ~1:00 pm

| Venues: | Breakfast – Santa Fe Room |
| | Morning and Panel Discussion - New Mexico Meeting Room |

|	
7:15 — 8:30	*Breakfast - provided by La Fonda on the Plaza*
8:30 — 9:10	Jyoti Mazumder
9:10 — 9:50	Suresh Babu
9:50 — 10:30	Kalman Migler
10:30 — 11:10	Christian Leinenbach
11:10 — 11:15	*Break with refreshments*
11:15 — ~1:00	Panel Discussion
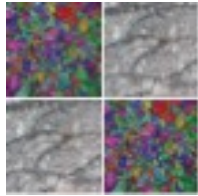
Dr. Pan Michaleris
Penn State University

Thermo-Mechanical Modeling of Additive Manufacturing Processes

Additive manufacturing (AM) has the potential to produce parts directly out of complex Computer Aided Design (CAD) files, which enables the direct manufacturing of geometries generated by topology optimization without the cost and delay of tooling as required for other processes such as forging and casting. However, distortion during part cooling negates the net shape potential of the process as overbuilding and follow on machining is commonly used to bring back parts into shape. Excessive distortion and part failure during manufacturing also commonly occurs causing process interruptions and part rejection. Due to the lack of effective and efficient modeling tools, the current approach to the design of AM processes is mostly experimental. In this work, thermo-mechanical modeling of AM processes is investigated to enable the virtual design and optimization of AM processing which will lead to reduced process design time, improved part performance, and reduced cost through a transition from an experimental-based to a model-based process design. Extensive model validation using *in situ* process monitoring has been performed on Direct Energy Deposition systems such as the Electron Beam Direct Manufacturing (EBDM) by Sciaky and the Laser Engineered Net Shaping System (LENS) by Optomec and model validation on powder-bed systems is currently underway.
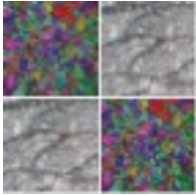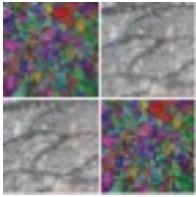
Hans-Joachim Schmid
Universität Paderborn

Controling Material Ageing as a Key Factor for
Product Quality in Polymer Laser Sintering

In polymer laser sintering only a small portion of the used powder is actually transformed into parts. Due to economical reasons, one would like to reuse the remaining powder in another build job despite the fact, that the polymer material is modified during the build process. This ageing of the polymer powder is in turn strongly influencing (i.e. deteriorating) resulting product properties. Therefore, one would typically use only a mixture of fresh powder with used powder. Hence, the amount of fresh powder needed to ensure a high product quality of laser sintered parts should be known as exact as possible.

In the first part of the talk, the effect of powder ageing on different product properties (mechanical, chemical, surface quality) is outlined and discussed. Subsequently, relevant ageing mechanisms of the polymer material and different methods to assess powder ageing are presented and discussed in detail. In the third part of the presentation detailed measurements of temperature history and ageing kinetics *inside* the build chamber are given. Furthermore a model to evaluate and predict local ageing kinetics is outlined and first results are presented. Aim of this work is to predict local powder ageing depending on process conditions and job layout in order to get a minimum powder refresh rate necessary to obtain best product quality. This appears to be vital for economical additive manufacturing of laser sintered parts.

Kevin Chou
The University of Alabama

Temperature Simulations and Measurements for Process Qualification in Powder-Bed Electron Beam Additive Manufacturing

Powder-bed electron beam additive manufacturing (EBAM) has a potential to offer innovative solutions to many challenges faced in the industry. However, the complex process physics of EBAM hinders efficient process development, qualification and optimizations, etc. Thermal characteristics such as temperatures and melt pools in powder-bed EBAM offer crucial process variable information that can be linked to the build part quality such as the accuracy and properties. To advance process qualification, a combined numerical-experimental approach was applied to research the process temperatures and melt pool sizes in EBAM using Ti-6Al-4V powder. The objective was to develop a comprehensive thermal model, using finite element analysis, for accurate temperature and melt pool predictions in EBAM. Simultaneously, a near infrared (NIR) thermal imager was attempted to acquire and analyze build surface temperatures in EBAM for model validations and thermal characteristic evaluations.

This seminar will detail the methodologies incorporated in thermal modeling and temperature measurements in EBAM. Challenges in the NIR thermography for EBAM such as the resolution, transmission loss, and emissivity estimate, etc., will be addressed. In addition, the research results such as temperature contours/profiles will be highlighted and compared including the process parameter effects and the influence of the powder-bed porosity. Future work to further process qualification in powder-bed metal additive manufacturing will also be discussed.
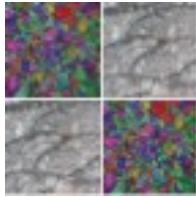
Mark D. Benedict
Air Force Research Laboratory

While Additive Manufacturing (AM) is beginning to deliver on the promise of revolutionizing the aerospace manufacturing industry, one of the largest barriers to broader adoption of AM for producing aerospace parts is the lack of a standardized qualification process. By leveraging investments with industry driven consortia such as the Metals Affordability Initiative and America Makes, the Air Force Research Laboratory is supporting research that enables the transition from slow and costly point qualifications to more general qualification and certification approaches. Key areas of targeted research that inform the qualification of additive materials and processes include: enabling fully characterized process definition, developing *in-situ* process monitoring and sensing, identifying the limits of AM part inspectability, and quantifying of the drivers of manufacturing variability in AM production parts. Recent progress made in three representative programs will be presented.
Case Number: 88ABW-2015-2948
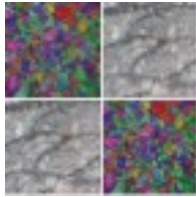
Natalie Rudolph
University of Wisconsin Madison

Experimental and Numerical Studies of the Temperature Field in Selective Laser Sintering to Improve Shrinkage and Warpage Prediction

Selective Laser Sintering (SLS) is among the most promising technologies for polymer additive manufacturing (AM) of small series. In this process a laser heat source locally melts polymer powder to fuse the material together permanently. This results in good mechanical performance compared to conventional bulk processing technologies such as injection molding. At the same time the still solid powder acts as a mold allowing for the most complex geometries and design freedom. In addition to the lack of material variety, another big challenge for SLS is the locally varying shrinkage and warpage of the parts, which is a challenge for all AM technologies.

In this presentation the underlying principle of shrinkage and warpage in AM will be discussed and examples for SLS, Fused Deposition Modeling (FDM) and Automated Fiber Placement (AFP) will be given. The focus of the talk will be on experimental and numerical studies of the temperature field in the SLS building chamber and preliminary results of a warpage simulation. The latter includes the coupling of the crystallization kinetics with the development of the modulus as a function of temperature and thus degree of crystallinity. This would eventually allow the better prediction of shrinkage and warpage as a function of the used machine (knowing the temperature distribution in the building chamber, laser power and speed and laser strategy), and process parameters (part distance to cylinder walls, part separation, part position in build job, layer time).

Finally, an in-situ measurement technique, Dielectrical Analysis (DEA), commonly used in composite manufacturing will be presented that allows the monitoring of crystallization during the process. The technique itself and first results will be presented, which are of interest to SLS but also to the currently new developments in the area of composite additive manufacturing.
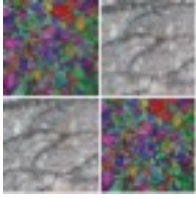
Dr. Christopher Tuck
University of Nottingham

Selective Laser Melting of Aluminum and Titanium Alloys - Some lessons learned

Selective laser melting is a powerful technique for the manufacture of complex structures in metallic materials. It is however, a method that is not well understood, this presentation outlines work that is ongoing at The University of Nottingham on the understanding of SLM from input materials; processing effects and understanding the final component's integrity. The talk will discuss issues of laser spatter, porosity measurement and analysis through the use of micro-CT techniques at Nottingham.
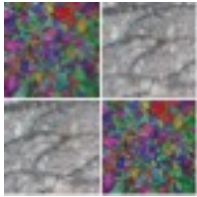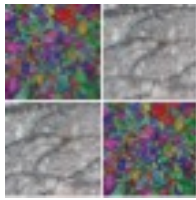
Speaker Abstracts

Dr. Chantal K. Sudbrack
NASA Glenn Research Center

Powder-bed fabrication of high temperature Ni-based superalloy LSHR

Additive manufacturing (AM) technologies may have the potential to revolutionize the production of aerospace components by eliminating the need for extensive machining or expensive legacy tooling. The scientific community has largely focused on developing an understanding of AM for aerospace alloys Ti-6A-4V and Inconel 718. Less-weldable advanced Ni-based γ'-superalloys, common to land-based and aviation gas turbines, can operate 500 °F hotter than Inconel 718 and represent the state-of-the-art for aerospace AM. Their superior high-temperature performance is mainly due to high refractory content (i.e. Mo, Nb, Ta, W) and stable intermetallic γ'-precipitates, both which make these alloys more prone to quench rate cracking during rapid solidification. Heated-bed electron beam melting (EBM) offers several distinct advantages over non-heated laser melting methods, including less retained residual stress, lower risk of contamination, slower cooling rates and faster build rates with multiple-electron-beam configurations. These advantages are attractive for turbine components, where such issues may shorten the high-temperature, long-range operation. The work herein demonstrates the feasibility of fabricating superalloy components using EBM AM through fabrication of low-solvus high-refractory (LSHR) superalloy, which is a turbine disk superalloy that contains ~55% γ'-content and 10 wt.% of refractory additions. The challenges to produce homogeneous builds with desired properties will be discussed, with particular attention to the effect of post-processing heat treatments on microstructure and placed into context of programmatic development.
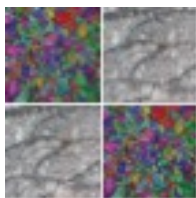
Speaker Abstracts

Todd Palmer
PennState University

Application of Fundamental Processing-Structure-Property Relationships in Accelerating Certification of Directed Energy Deposition Processes

Directed energy deposition processes in additive manufacturing (AM) utilize a high energy density heat source, such as a laser or electron beam, to melt either injected powder or wire in a layer-by-layer manner to produce discrete structural components. The resulting heat source-material interactions produce a range of complex processing conditions, which include rapid melting and solidification and multiple solid state phase transformations. These complex processing conditions, in turn, produce a range of microstructures both within individual builds and across different processing conditions, leading to high levels of variation and uncertainty in mechanical properties and performance characteristics. A key component to developing certification protocols for AM components is a fundamental understanding of these complex processing-structure-property relationships which minimize the level of uncertainty in the resulting material properties. Current work on the development of these relationships in both nickel and titanium alloy systems is reviewed, along with how they are being applied to current certification protocols.

Lyle Levine
NIST[1]

From Powder to Parts: Making Sense of Metals Additive Manufacturing using Modeling, *In Situ* Monitoring, and *Ex Situ* Characterization

Additive manufacturing (AM) of metal components is a rapidly expanding manufacturing paradigm that could revolutionize the design and production of complex, high-value metal parts. However, the extreme processing conditions create inhomogeneous materials that may include varying metallic phases, voids, and localized stresses. With a goal of optimizing the build and post-build processing of these complex materials, we are combining state-of-the-art, *in situ* thermography; finite element modeling (FEM); comprehensive *ex-situ* microstructural characterization using world-leading synchrotron facilities and state-of-the-art lab-based measurements; and multicomponent computational thermodynamic and kinetic microstructural evolution simulations. For example, thermography is used to monitor the temperature distributions during the build process and to validate FEM predictions. FEM-based "design of experiment" studies are then used to investigate the sensitivity of the build process to numerous process parameters. FEA also provides a thermal history for conducting detailed microstructural evolution simulations of the build and post-build (annealing) processes. These microstructure models are validated using synchrotron and lab-based studies such as microbeam X-ray diffraction that is used to obtain local (submicrometer) stress tensors and three-dimensional maps of crystallographic phases and orientations. Ultra-small-angle X-ray scattering (USAXS), SAXS and wide-angle X-ray scattering (WAXS) are used together to monitor the microstructural evolution during post-production annealing. Finally, mechanical testing is carried out to evaluate the mechanical behavior of the manufactured test specimens. This talk will primarily concentrate on AM-produced Ni-based super alloys.

Carelyn Campbell[1], Li Ma[1], Brandon Lane[2], Yaakov Idell[1], Eric Lass[1], Fan Zhang[1], Andrew Allen[1], Mark Stoudt[1], Sudha Cheruvathur[1], Shawn Moylan[2], Ruqing Xu[3], and Jan Ilavsky[3]

[1]Material Measurement Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA. [2]Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA. [3]Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439-4800, USA.
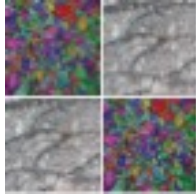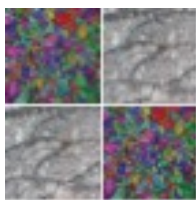
Jyoti Mazumder
University of Michigan at Ann Arbor

A Challenge for Additive Manufacturing: "Certify as you build"

Although, additive Manufacturing (AM) has been hailed as the "third industrial revolution" by The Economist magazine [April-2012], many of the key patents are either expired or about to expire. For example, the first patent on Stereo-lithography was awarded in 1986. AM is an enabling technology which can build, repair or reconfigure components layer by layer or even pixel by pixel with appropriate materials to match the performance will enhance the productivity thus reduce energy consumption. Innovative product such a metallic composites with negative co-efficient of thermal expansion has already been demonstrated. The capability to form a three-dimensional object directly form digital data reduces many intermediate steps in manufacturing and, therefore, potentially an attractive and economic fabrication method. This is very suitable for low volume manufacturing. Moreover, U.S. Industry loses about $57 Billion per years due to quality control issues (Forbes, 2006). The major challenge for adaptation of additive manufacturing is on line "Quality assurance", due to its application in low volume manufacturing. Statistical quality control is not applicable due to low volume. Moreover, any new innovative product has to go thorough a long process of certification before adaptation, especially for Aerospace and medical device industry.

This paper presents the methodology for Smart Metallic Additive Manufacturing System (s-AMS). In-situ optical diagnostics and its capability to integrate with the process control is a prudent alternative. The two main groups of AM are powder bed (e.g. Laser Sintering) and pneumatically delivered powder (e.g. Direct Metal Deposition [DMD]) to fabricate components. DMD enables one to deposit different material at different pixels with a given height directly from a CAD drawing. The feed back loop also controls the thermal cycle. New optical Sensors are being developed to control product health and geometry using imaging, cooling rate by monitoring temperature, microstructure and composition using optical spectra. Ultimately these sensors will enable one to "Certify as you Build". Recently the author and his group have developed a technique to analyze the plasma spectra to predict the solid-state phase transformation, which opens up the new horizon for the materials processing and manufacturing. Mathematical model developed for the process includes most of the physics but need substantial computing time. An effort needs to be made to develop surrogate models, which can converge within 10ms to enable the process control.

Flexibility of the process is enormous and essentially it is an enabling technology to materialize many a design. Conceptually one can seat in Santa Fe and fabricate in Sheffield. This paper discusses the in-situ diagnostic methods and its integration in the additive manufacturing process.

Suresh S. Babu
University of Tennessee

Innovative Process Controls and Qualification of Additively Manufactured Metallic Components with Tailored Microstructures and Properties
Suresh S. Babu, R. Dehoff, L. Love and W. Peter

Additive manufacturing of metals is considered as a disruptive technology to produce limited number of high value components with topologically optimized geometries and functionalities. Realization of the above potential for real-world applications is stifled by lack of standard qualification of computational design-tools; material feed stock characteristics, methods to probe thermo-mechanical processes under in-situ conditions, and microstructural homogeneity, as well as, anisotropic static- and dynamic-properties. This presentation will discuss the needed interdisciplinary science and technology ranging from robotics and automation, process control, multi-scale in-situ and ex-situ characterization methodologies, as well as, high-performance computational tools to address these challenges. Specific focus on understanding and controlling physical processes will be stressed, including powder/wire/tape, powder sintering, adsorption and dissolution of gases, microstructure evolution under extreme thermal gradients, and residual stress evolution under complex thermal gyrations. Case studies demonstrating the usefulness of these strategies will be presented. Emerging pathways to scale up metal additive manufacturing) in Fe-, Al- and Ti- alloys to large sizes (>1 m) and higher productivity (5 to 20 kg/h), while maintaining the mechanical performance and geometrical flexibility expected by the additive manufacturing, will be discussed.
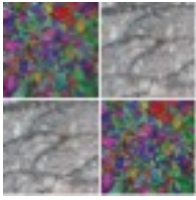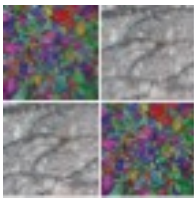
Speaker Abstracts

Kalman Migler
NIST

Enabling Qualification of FFF via Fundamental Process Measurements

Fused Filament Fabrication (FFF) is a compelling Additive Manufacturing technology because of the wide variety of thermoplastics it can utilize. However, the specific extrusion and joining processes employed by FFF are conducted under vastly different thermal and flow profiles compared to traditional processing. This leads to uncertainties in how to optimize the materials or the machine parameters FFF, and limits development of strategies on how to improve final part properties, which are typically reduced compared to traditional polymer processing. Our approach is to provide guidance through the great "phase space" of materials, mixtures, and processing protocols by development of the tools that link the macro-scale kinetics with those at the molecular scale.   We present preliminary results that illuminate this approach through measurement of thermal profiles (through IR thermography), the subsequent interdiffusion of polymer between layers (neutron reflectivity) and mechanical properties. These measurements provide a basis from which in-situ measurements for QC can be obtained. We also present results showing feasibility of using Raman spectroscopy for on-line measurements of material composition, an essential element of Qualification.

Christian Leinenbach
Empa, Swiss Federal Laboratories for Materials Science & Technology

Meta Materials Aspects in Metal Additive Manufacturing –
Challenges, Opportunities, Visions

Metal additive manufacturing (AM) techniques are powder-based, layer by layer methods which can directly build 3D structures onto substrates with complex geometries. They offer a unique ability to dynamically mix materials during the deposition process and produce functionally graded structures, new composite microstructures and perhaps even new material classes. An important benefit lies also in the possibility of repairing components. For the additive manufacture of metals and alloys, selective laser melting (SLM), selective e-beam melting (EBM) and laser direct metal deposition (LMD) are usually applied. However, there have been only a limited number of more or less easily processable alloys used in SLM so far, and the processing parameters are usually obtained from a trial-and-error approach. In order to exploit the advantages of SLM, novel alloys and composites those are adapted to the special processing conditions during SLM need to be developed. This requires a deep understanding of the materials science of metal additive manufacturing, which is currently still lacking.

Some of the challenging issues related to the energy beam based process are the very high heating and cooling rates, leading to non-equilibrium microstructures, which are usually harder, less ductile, and often exhibit high residual stresses; the strongly textured, anisotropic microstructures inherited from the solidification conditions; or the pronounced residual stresses resulting from the large thermal gradients in the AM fabricated parts.

However, the very rapid consolidation of the material in a small material volume and the achieved high solidification rates allow for the manufacture of components containing meta-stable materials, as for example metal-diamond composites or new types of oxide dispersion strengthened (ODS) alloys, which are difficult or even impossible to manufacture using conventional casting or sintering processes.

This talk will give an overview of the challenges and opportunities in metal additive manufacturing from a materials scientist's perspective and some relevant results of the AM related research at Empa will be presented. Finally, some visions for future AM concepts regarding in-situ process monitoring and control will be discussed.

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CONSORTIUM

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Veronica Livescu

Image:
Rusty Gray